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RADIOCHEMICAL DETERMINATION OF CCRMP REFERENCE MATERIALS FOR CERTIFICATION PURPOSES: LEAD-210 ACTIVITY MEASUREMENTS

FINAL REPORT

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BY

UNIVERSITY OF WATERLOO

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MICROMEDIA

RADIOCHEMICAL DETERMINATION OF CCRMP REFERENCE MATERIALS FOR CERTIFICATION PURPOSES: LEAD-210 ACTIVITY MEASUREMENTS

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The lead-210 content in the compositional reference materials DL-4a, DH-4a, BL-4a and BL-5 supplied by CANMET, Mineral Sciences Labs, 555 Booth Street, Ottawa, Ontario K1A 0G1 has been determined by radioanalytical methods. The results are presented in Table 1 and the methodology is described below.

Weighed amounts of each reference material (in quadruplicate) were dissolved in a mixture of nitric, perchloric and hydrofluoric acids at 150^{o}C in a teflon bomb. The dissolved mass was transferred to a teflon beaker and evaporated to dryness. The dried mass was dissolved in concentrated hydrochloric acid and the solution was again evaporated to dryness. It was then brought to 1M HCl and a small amount of 0.17M solution of diethylenetriaminepentaacetic acid added. The solution was boiled for one minute and cooled. Sufficient amount of ascorbic acid was added to reduce Fe(III) to Fe(II). Lead was extracted with a solution of diethlyammonium diethlydithiocarbamate (DDTC) in xylene. The extract after washing, was plated on a planchet. Sufficient care was taken so that the sample was deposited evenly on the planchet. Aliquots of the standard solution of lead-210 supplied by the CANMET laboratory

were similarly treated so as to enable determination of chemical recovery by the chemical procedure. The recovery of lead by the extraction procedure with DDTC was found to be ∿ 99.9 per cent.

1-ml aliquots of the standard solution were deposited on planchets and the activity due ^{210}Pb - ^{210}Bi was assayed in each aliquot with a low-background gas flow proportional counter with ~ 5 cm diameter window. The planchets were covered with a thin absorber to ensure that alpha particles from ^{210}Po, if present in the sample, did not reach the counter. The total accumulated counts in each sample exceeded 20,000 with the background as ∿ 100 counts per hour. The counting time for the lead-210 samples separated from the standard materials was adjusted according to the count rate. In most cases, the total accumulated counts exceeded 20,000 counts. The counting efficiency of the counter was found to be 0.243 ± 0.001 count per disintegration. Another standard source obtained from the New England Nuclear (NEN) was also used for the determination of the efficiency of detection. The two values agreed within the expected error. 1-ml pipette used for plating the standard solution had a calibration accuracy of 0.006 ml or the measurement of efficiency of detection can be considered to have a systematic error of 0.6 per cent.

The standard deviation, σ as given in Table 1, has been evaluated from the counting data as square root of total counts for the sample. The per centage error as quoted is equal to

$\frac{(\text{total counts})^{\frac{1}{2}} \times 100}{\text{total counts - background}}$. The 95% confidence level can be taken as 2σ. Additional error of 0.5 per cent in the determination of the detection efficiency is not included in the σ value.

There are two comments that can be made concerning the methodology and the measurements.

a) The dissolution of Reference material BL-4a was not complete. However, there were very few β counts observed in the residue. It appears that there was hardly any Pb-210 present in the residue.

b) The β counting introduces additional uncertainty in the measurements. The lead-210 samples were carefully deposited on planchets by evaporation of liquid under a heat lamp. The technique does not ensure uniform deposition over the entire area. The measurements do have uncertainties due to self absorption in the sample and due to backscattering from the planchet. It was not possible to prepare weightless samples deposited on a gold-coated thin film in our laboratory and to measure beta particles from the sample by a 4π windowless gas-flow proportional counter for the estimation of Pb-210.

TABLE 1

Lead-210 Content in CCRMP Reference Materials

Sample	Sample submass for radiochemical assay in gram	Activity in Bg/g	Standard deviation, σ	% age error
DL-1a	1.2199	1.46	0.012	0.78
	1.3200	1.39	0.014	0.99
	0.9000	1.33	0.011	0.79
	1.1506	1.35	0.013	0.94
BL-4a	1.2501	15.17	0.13	0.82
	1.2650	14.22	0.11	0.79
	1.2605	14.64	0.12	0.79
	1.0495	14.37	0.13	0.87
DH-1a	1.0001	30.4	0.19	0.61
	1.1002	29.8	0.20	0.65
	0.9720	29.5	0.19	0.63
	1.0300	29.0	0.18	0 62
BL-5	0.0960 (a)	848	3.2	0.38
	0.1150	894	3.0	0.33
	0.0910	812	3.2	0.39
	0.1180	860	2.9	0.34

(a) aliquot taken for the Pb-210 determination